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| 09/403,224 | 10/15/1999 | KOJI MATSUMOTO | 0020-4621P | 6995 |

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EXAMINER

WILSON, DONALD R

ART UNIT

PAPER NUMBER

1713

DATE MAILED: 12/21/2001

Please find below and/or attached an Office communication concerning this application or proceeding.

T-D - 8

Office Action Summary

Application No.

09/403,224

Applicant(s)

MATSUMOTO, KOJI

Examiner

D. R. Wilson

Art Unit

1713

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☒ Responsive to communication(s) filed on 21 November 2001.
- 2a) ☒ This action is FINAL. 2b) ☐ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 1-8 is/are pending in the application.
- 4a) Of the above claim(s) 8 is/are withdrawn from consideration.
- 5) ☐ Claim(s) _____ is/are allowed.
- 6) ☒ Claim(s) 1-7 is/are rejected.
- 7) ☐ Claim(s) _____ is/are objected to.
- 8) ☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on _____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
- 11) ☐ The proposed drawing correction filed on _____ is: a) ☐ approved b) ☐ disapproved by the Examiner.
If approved, corrected drawings are required in reply to this Office action.
- 12) ☐ The oath or declaration is objected to by the Examiner.

Priority under 35 U.S.C. §§ 119 and 120

- 13) ☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
a) ☒ All b) ☐ Some * c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. _____.
3. ☒ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).
* See the attached detailed Office action for a list of the certified copies not received.
- 14) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. § 119(e) (to a provisional application).
a) ☐ The translation of the foreign language provisional application has been received.
- 15) ☐ Acknowledgment is made of a claim for domestic priority under 35 U.S.C. §§ 120 and/or 121.

Attachment(s)

- 1) ☐ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO-1449) Paper No(s) _____
- 4) ☐ Interview Summary (PTO-413) Paper No(s). _____
- 5) ☐ Notice of Informal Patent Application (PTO-152)
- 6) ☐ Other: _____

DETAILED ACTION

Election by Original Presentation

1. Newly submitted Claim 8 is directed to an invention that is independent or distinct from the invention originally claimed for the following reasons: Claim 8 is a second group of inventions related to the first group of inventions as (i) process of making and product made, or (ii) as product and process of use. The inventions are distinct if the product as claimed can be made by another and materially different process (MPEP § 806.05(f)), or can be used in a materially different process of using that product (MPEP § 806.05(h)). In the instant case the product as claimed can be made by another and materially different process such as by curing with peroxides other than claimed, and can be used in a materially different process of using that product such as a coating.

2. The inventions of the two groups do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical feature(s), or the special technical feature(s) does not define a contribution over the prior art for the reasons which have been set forth in the prior art rejections. Accordingly, the special technical feature linking the two inventions does not provide a contribution over the prior art.

3. Since applicant has received an action on the merits for the originally presented invention, this invention has been constructively elected by original presentation for prosecution on the merits. Accordingly, Claim 8 is withdrawn from consideration as being directed to a non-elected invention. See 37 CFR 1.142(b) and MPEP § 821.03.

Response to Amendment

4. Applicant's amendment filed 11/21/01, has been fully considered with the following results.

5. The amendment has overcome the rejection under 35 U.S.C. § 112, second paragraph, in regards to (i) the amounts of acetone and tert.-butanol decomposition products, and (ii) the ambiguous meaning of "primarily" and "secondarily" cured products, and in this regard the rejection is withdrawn. However, the amendment is not deemed to be persuasive in overcoming the remaining issue as is discussed below.

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6. The amendment is not deemed to be persuasive in overcoming the prior art rejections which are maintained for the reasons discussed below.

Previously Cited Statutes

7. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claim Rejections - 35 USC § 112, Second Paragraph

8. ***Claims 6-7 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.*** The basis of this rejection was stated in Detailed Action § 4 of the previous Office Action.

9. Claims 6 and 7 are indefinite because the contribution of secondary curing to a compression set value would be dependent on the conditions of primary curing as well as the conditions of secondary curing, none of which is specified in the claim. Applicant has not traversed the basis of this rejection which remains valid.

Claim Rejections - 35 USC § 102(b) - 35 USC § 103

10. ***Claims 1-7 are rejected under 35 U.S.C. 102(b) as anticipated by or, in the alternative, under 35 U.S.C. 103(a) as obvious over WO'995, Takemoto, or Albano.*** The basis of this rejection was stated in Detailed Action § 9-12 of the previous Office Action.

11. Applicant traverses the rejection arguing that none of the references "--- disclose or suggest organic peroxides in the amounts recited in the claims" and that the curable compositions "--- can achieve unexpected superior results of (sic) over the cited references". This is not deemed to be persuasive because each of the references have specific disclosures of organic peroxides and amounts which are within those of the instant claims. Thus, WO'995 specifically discloses the use of t-butyl cumyl peroxide and dicumyl peroxide (page 3, lines 9-13), and the use of 0.3 parts by weight (pbw) of the peroxide is also specifically taught (page 3, lines 17-19, and Claim 4). The use of peroxide in amounts of 0.5 and 1 pbw is exemplified in Examples 1 and 2. Thus, one would have readily envisaged the use of t-butyl cumyl peroxide or dicumyl peroxide in amounts of 0.3, 0.5 and 1.0 pbw in the compositions of WO'995. Takemoto specifically discloses the use of di-t-butylperoxide, t-butycumyl-peroxide and dicumylperoxide

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(col. 4, line 62 to col. 5, line 10), and the preferable use of 0.5 pbw is specifically taught (col. 5, lines 24-26). Thus, one would have readily envisaged the use of di-t-butylperoxide, t-butyl cumyl peroxide or dicumyl peroxide in amounts of 0.5 pbw in the compositions of Takemoto. Albano specifically discloses curing with di-t-butylperoxide, and dicumylperoxide (col. 4, line 64 to col. 5, line 6), and the use of 0.5 and preferably 1 % by weight is specifically taught (col. 2, lines 54-56). Thus, one would have readily envisaged the use of di-t-butylperoxide, or dicumyl peroxide in amounts of 0.5 and 1.0 wt.% in the compositions of Albano. In regards to the unexpected superior results, this would overcome the obviousness aspect of the rejection, but the claims are still deemed to be anticipated in regards to the peroxides present and their amounts. As to limitations of the total amount of acetone and t-butanol in the decomposed product of the peroxides, this would be an inherent property as the peroxides are the same as those in applicant's inventive compositions.

12. In regards to the molded products (Claims 5-6), limitations of the amount of decomposed products of the peroxide, or contribution of a compression set during secondary curing is not seen to distinguish the molded articles over those taught and/or obvious from the teachings of the references, as these are product-by-product limitations. For instance, it is not seen that the properties of the modified articles in the instant comparative examples after secondary curing are patentably distinguishable from those of the inventive samples after the primary curing.

Action Is Final

13. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

14. A shortened statutory period for reply to this final action is set to expire **THREE MONTHS** from the mailing date of this action. In the event a first reply is filed within **TWO MONTHS** of the mailing date of this final action and the advisory action is not mailed until after the end of the **THREE-MONTH** shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than **SIX MONTHS** from the date of this final action.

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15. This application contains Claim 8 drawn to an invention nonelected by original presentation. A complete reply to the final rejection must include cancelation of nonelected claims or other appropriate action (37 CFR 1.144) See MPEP § 821.01.

Other Comments

16. Machine translations of JP'776, JP'391 and JP'879 each cited in the international search report as "X" references, and mentioned in the previous Office Action, are attached. It appears that the references are at best cumulative to those already applied.

17. Applicant's request for an interview is noted. However, the time to request an interview is before the Examiner is in the process of acting on applicant's response. If applicant still wishes to have an interview, they are invited to make an appointment by telephoning the Examiner.

Future Correspondence

Any inquiry concerning this communication or earlier communications from the examiner should be directed to D. R. Wilson whose telephone number is 703-308-2398.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, David Wu can be reached on 703-308-2450. The fax phone numbers for the organization where this application or proceeding is assigned are 703-305-5408 for regular communications and 703-305-3599 for After Final communications. The unofficial direct fax phone number to the Examiner's desk is 703-872-9029.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the receptionist whose telephone number is 308-2351.



D. R. Wilson
Primary Examiner
Art Unit 1713

* NOTICES *

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~~09-059391~~
JP 09/183 879

1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] (A) The fluororubber constituent which comes to contain the 4th class ammonium hydrogensulfate of (B) organic and/or 1, the salt 0.1 guided from 8-diazabicyclo [5.4.0]-7-undecene - 10 weight section, (C) metallic oxide and/or the metal hydroxide 0.5 - 30 weight section, (D) organic peroxide 0.5 - 20 weight section, (E) polyfunctional nature compound 0.5 - 20 weight section, and the phenolic compound 0.1 which has (F) electron withdrawing substituent - 20 weight section to the fluororubber 100 weight section.

[Translation done.]

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the new fluororubber constituent with which vulcanization nature has been improved still in detail about a fluororubber constituent.

[0002]

[Description of the Prior Art] Since a fluororubber has the outstanding thermal resistance, oil resistance, chemical resistance, etc., it is widely used in various kinds of industrial fields focusing on autoparts etc. The fluororubber material with more high thermal resistance is demanded by elevation of the engine temperature especially produced from the high increase in power of an engine in recent years. As a material which fills this demand, the organic peroxide vulcanization type fluororubber constituent attracts attention as a material which fills these demands. For example, in JP,1-198654,A, the constituent for fluororubber vulcanization, which becomes (a) ** fluorine elastic copolymer from (b) organic peroxide, (c) polyfunctional nature compound, and a divalent metal hydroxide and/or a divalent metallic oxide and (d) (e) organic base is proposed. However, a scorching stability is inferior, and if kneading ***** is high at a roll, a Banbury mixer, etc. in the kneading ***** case, vulcanization torque will fall, and this constituent has the problem that a compression set becomes large further.

[0003] Moreover, the effect is not enough for it although the fluororubber constituent which consists of the fluororubber which consists of a (A) fluoride vinylidene and an ethylene nature unsaturated hydrocarbon, (B) organic peroxide, a divalent metal hydroxide or a divalent metallic oxide and (C) (D) organic base and a (E) phenol system compound, or an aromatic-amine system compound is proposed by JP,7-179704,A.

[0004]

[Problem(s) to be Solved by the Invention] this invention was made against the background of the technical problem of the above-mentioned conventional technique, a scorching stability and its stability of the vulcanization behavior to kneading ***** are good, and it aims at offering the good fluororubber constituent of a mechanical property.

[0005]

[Means for Solving the Problem] The salt 0.1 with which this invention is guided to the (A) fluororubber 100 weight section from 4th class ammonium hydrogensulfate [of (B) organic] and/or 1, and 8-diazabicyclo [5.4.0]-7-undecene, - 10 weight section, (C) A metallic oxide and/or the metal hydroxide 0.5, - 30 weight section, (D) The fluororubber constituent which comes to contain organic peroxide 0.5 - 20 weight section, (E) polyfunctional nature compound 0.5 - 20 weight section, and the phenolic compound 0.1 which has (F) electron withdrawing substituent - 20 weight section is offered.

[0006]

[Embodiments of the Invention] The characteristic feature of this invention is that it blended with the fluororubber the salt guided as a vulcanization accelerator from 4th class ammonium hydrogensulfate [of organic] and/or 1, and 8-diazabicyclo [5.4.0]-7-undecene, and organic peroxide and a polyfunctional nature compound, a metallic oxide and/or a metal hydroxide and the phenolic compound that has an electron withdrawing substituent.

[0007] (A) The fluororubbers of a component are a fluoride vinylidene and a copolymer which carries out copolymerization of at least one sort of ethylenic unsaturated monomers which can be copolymerized to this, and is obtained. Here, as an ethylenic unsaturated monomer, hexafluoropropylene, a pentafluoro propylene, triffe ***** ethylene, a trifluorochloroethylene, a tetrafluoroethylene, the vinyl full ora id, perfluoro (methyl vinyl ether), perfluoro (propyl vinylidene), a propylene, etc. can be mentioned.

[0008] (A) As an example of a fluororubber, the tetrafluoroethylene-fluoride vinylidene-propylene ternary-polymerization field, the tetrafluoroethylene-fluoride vinylidene-hexafluoropropylene ternary-polymerization field, a fluoride vinylidene-hexafluoropropylene copolymer, etc. are mentioned, and the tetrafluoroethylene-fluoride vinylidene-propylene ternary-polymerization field is especially desirable. For a fluoride vinylidene unit, in the tetrafluoroethylene-fluoride vinylidene-propylene ternary-polymerization field, a 2-70 mol % and tetrafluoroethylene unit is [the 20-60 mol % and propylene unit of the desirable domain of each monomer] 20-60 mol % (however, fluoride vinylidene unit + tetrafluoroethylene unit + propylene unit = 100 mol %). For a fluoride vinylidene unit, a 3-60 mol % and tetrafluoroethylene unit is [the 20-60 mol % and propylene unit of a still desirable domain] 20-60 mol %.

[0009] ** et al. -- ** -- (A) fluororubber is independent, or two or more sorts are mixed and it is used Moreover, especially the molecular weight or molecular weight distribution of these fluororubbers are not restricted, and are suitably chosen according to

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intended use, a process condition, etc. The polymerization method with conventionally well-known an emulsion polymerization, a suspension polymerization, solution polymerization, a bulk polymerization, etc. is preferably adopted as a manufacture of these (A) fluororubbers.

[0010] (B) component used in this invention promotes a ** hydrogen fluoride reaction from a fluororubber at the time of vulcanization, and has the operation which makes it vulcanize smoothly. This (B) component is a salt guided from 4th class ammonium hydrogensulfate [of organic] and/or 1, and 8-diazabicyclo [5.4.0]-7-undecene. the example Sulfuric-acid hydrogen tetrabutyl ammonium, sulfuric-acid hydrogen trioctyl methylammonium, Sulfuric-acid hydrogen benzyl trimethylammonium, sulfuric-acid hydrogen 1, 8-diazabicyclo [5.4.0]-7-***** chloride, Sulfuric-acid hydrogen 1, a 8-diazabicyclo [5.4.0]-7-***** star's picture, The 8-methyl -1, 8-diazabicyclo [5.4.0]-7-***** chloride, The p-toluenesulfonic-acid salt of 8-methyl -1, 8-diazabicyclo [5.4.0]-7-***** star's picture, 1, and 8-diazabicyclo [5.4.0]-7-undecene etc. is mentioned. The thing to use by the one sort independent can also be mixed and used for these compounds for two or more sorts.

[0011] (B) the amount of the component used -- the (A) fluororubber 100 weight section -- receiving -- 0.1 - 10 weight section -- desirable -- 0.2 - 7 weight section -- it is 0.5 - 5 weight section still preferably Under in 0.1 weight section, if a sufficient vulcanization density and sufficient physical properties are not acquired but 10 weight section is exceeded on the other hand, while fabricating-operation nature, such as scorching development nature, will be affected, the property of which chemical-resistant vulcanizate is reduced.

[0012] All the well-known compounds that (C) metallic oxide and/or the metal hydroxide which are used for this invention are used as carrier acid at the time of vulcanization of a fluororubber, and are used at the time of polyol vulcanization are conventionally usable. (C) As an example of a component, a magnesium oxide, a calcium oxide, a zinc oxide, a lead oxide, a calcium hydroxide, a magnesium hydroxide, etc. are mentioned, and the thing to use by the one sort independent can also mix two or more sorts, and can also use these compounds.

[0013] (C) the amount of the component used -- the (A) fluororubber 100 weight section -- receiving -- 0.5 - 30 weight section -- desirable -- 1 - 25 weight section -- it is 2 - 10 weight section still preferably Under in 0.5 weight section, if a sufficient vulcanization density and sufficient physical properties are not acquired but 30 weight section is exceeded on the other hand, an intensity will fall.

[0014] (D) organic peroxide used for this invention is an organic compound which is used as a vulcanizing agent and has combination (-O-O-). As an example of organic peroxide, (D) Benzoyl peroxide, Dicumyl peroxide, 2, the 5-dimethyl -2, 5-di-tert-butyl peroxide hexane, 2, the 5-dimethyl -2, 5-di-tert-butyl peroxide hexyne -3, a G (tert-butyl peroxide)-m-G isopropylbenzene, 1 and 1-screw (tert-butyl peroxide) - 3, 3, a 5-trimethyl cyclohexane, alpha and alpha'-screw (tert-butyl peroxide)-p-diisopropylbenzene, G t-butyl peroxide, t-butyl par oxybenzoate, 2, 4-dichloro benzoyl peroxide, p-***** benzoyl peroxide, etc. are mentioned, and the thing to use by the one sort independent can also be mixed and used for these organic peroxide for two or more sorts.

[0015] (D) the amount of the component used -- the (A) fluororubber 100 weight section -- receiving -- 0.5 - 20 weight section -- desirable -- 1 - 10 weight section -- it is 1 - 5 weight section still preferably Under in 0.5 weight section, if a sufficient vulcanization density and sufficient physical properties are not acquired but 20 weight section is exceeded on the other hand, physical properties, such as fracture elongation, will fall.

[0016] (E) polyfunctional nature compound used for this invention is used as a vulcanization assistant, and many allyl compounds, a methacrylate compound, a ***** compound, a bismaleimide compound, an oxime compound, etc. are mentioned. As an example of a polyfunctional nature compound, (E) Triallyl isocyanurate, Trimethylolpropanetrimethacrylate, N, and N'-m-phenylene bismaleimide, Ethylene glycol dimethacrylate, 1, 3-butanediol ***** acrylate, 1, 4-butanediol ***** acrylate, 1, 6-hexanediol dimethacrylate, Poly-ethylene glycol dimethacrylate, 1, 4-butanediol diacrylate, A 1, 6-hexanediol diacrylate, 2, and 2'-screw (4-methacryloyl diethoxy phenyl) propane, Trimethylolpropane triacrylate, a pentaerythritol thoria chestnut rate, A divinylbenzene, N, and N'-methylene screw acrylamide, p-quinonedioxime, p and p'-dibenzoyl quinonedioxime, a triazine dithiol, A triaryl ***** rate, bismaleimide, etc. are mentioned and it is triallyl isocyanurate, trimethylolpropanetrimethacrylate, N, and N'-m-phenylene bismaleimide preferably. The thing to use by the one sort independent can also be mixed and used for these (E) polyfunctional nature compounds for two or more sorts.

[0017] (E) the amount of the component used -- the (A) fluororubber 100 weight section -- receiving -- 0.5 - 20 weight section -- desirable -- 1 - 10 weight section -- it is 1 - 8 weight section still preferably Under in 0.5 weight section, if a sufficient vulcanization density and sufficient physical properties are not acquired but 20 weight section is exceeded on the other hand, vulcanizate nature, such as elongation, will fall.

[0018] As an electron withdrawing substituent of the phenolic compound which has (F) electron withdrawing substituent used for this invention, a nitrile group, a carboxyl group, a nitro group, a carbonyl group, a halogen machine, a phenyl group, and the base that replaced these parts by other bases are mentioned. As for the position of this electron withdrawing substituent, what is in the ortho position and the para position to the hydroxyl group of a phenolic compound is desirable. Moreover, it is more desirable not to include a substituent except an electron withdrawing substituent in the ring of a phenol. As a phenolic compound which has concrete (F) electron withdrawing substituent, o-phenylphenol, p-phenylphenol, ethyl para hydroxybenzoate, a para-hydroxybenzoic-acid isobutyl, a para-hydroxybenzoic-acid isopropyl, etc. are mentioned. The thing to use by the one sort independent can also be mixed and used for these (F) compounds for two or more sorts.

[0019] (F) the amount of the component used -- the (A) fluororubber 100 weight section -- receiving -- 0.1 - 20 weight section --

desirable -- 0.1 - 10 weight section -- it is 0.1 - 5 weight section still preferably

[0020] It is possible to blend bulking agents, such as other components, many carbon black, a silica, clay, the diatom earth, a calcium carbonate, a calcium fluoride, and a barium sulfate, a reinforcing agent, processing aid, an internal release agent, an adhesion promoter, a plasticizer, a coloring agent, etc. with the fluororubber constituent of this invention if needed. Moreover, it is also possible to blend natural rubber, other synthetic rubber, thermoplastics, thermosetting resin, etc.

[0021] The fluororubber constituent of this invention is obtained by mixing each above-mentioned component uniformly by kneading ***** of usual rubber, such as a roll and a kneader. Thus, the fluororubber constituent obtained can be fabricated completely like usual molding using metal mold, such as a press forming, injection molding, and transfer molding.

[0022] Moreover, the fluororubber constituent of this invention can also be vulcanized and fabricated by the molding technique of usual rubber, such as extrusion molding, calender molding, and coating after melting to a solvent, a dip forming. Although vulcanization conditions are suitably decided according to the configuration and conditions of a thing which it is going to fabricate, the domain of them is usually seconds [several] - 24 hours at 100-400 degrees C. Moreover, in order to stabilize the property of the vulcanizate obtained, you may perform secondary vulcanization. The conditions of this secondary vulcanization are usually 30 minutes - about 48 hours at 150-300 degrees C.

[0023] The vulcanizate which vulcanized the fluororubber constituent of this invention has the outstanding thermal resistance, oil resistance, weatherability, a compression set, and compressive-load-proof nature, and use in general industry, the electrical and electric equipment, and a chemistry field is possible for it.

[0024]

[Example] Although an example is given and this invention is explained still concretely hereafter, this invention is not limited to the following examples. In addition, among an example, the section and especially % are weight criteria, unless it refuses.

Moreover, it asked for the vulcanizate behavior and the tension test in an example, the compression-set examination, and the thermal stability of a compound as follows.

[0025] It measured with 170 degrees C and one amplitude using the product made from vulcanizate behavior Japanese ***** and JSR curelast meter V type.

A tension test, compression-set examination JIS It measured based on K6301.

The thermal-stability Mooney viscosity of a compound was judged in 121 degrees C for preheating 1 minute from the difference for measurement 4 minutes and (ML 1+4 and 121 degrees C), and measurement 10 minutes (1+10, 121 degrees C of MLs).

[0026] Furthermore, each component used for the example and the example of a comparison is as follows.

The tetrafluoroethylene / fluoride vinylidene / propylene (mole ratio) = 53 which it comes to obtain by the fluororubber emulsion-polymerization method / 5/42, the product made from fluororubber MT Asahi Carbon of intrinsic-viscosity $[\eta] = 0.55$, the product made from Asahi thermal MgO#150 consonance ****, a magnesium oxide [0027] the product made from cull Bit Omi Chemistry, and calcium-hydroxide TAIC triallyl isocyanurate TBAHS -- the product made from sulfuric-acid hydrogen tetrabutyl ammonium parka ***** 14-ized medicine ***** and a G tert-butyl peroxide-G isopropylbenzene [0028]

Composition of a compound (I) shown in one to examples 1-3 and example of comparison 2 tables 1-2 was *****ed for 5 minutes by rotational frequency 300rpm using the ***** leo code mixer (300 cc of inner capacities) with a setting temperature of 60 degrees C. Subsequently, compound shown in Tables 1-2 to the obtained compound (I) (II) It *****s for 5 minutes with the 4 inch roll which set composition as 50 degrees C and 80 degrees C, and is a compound (II). The constituent was obtained. This compound (II) After vulcanizing 2mm thick sheet and the test piece for compression-set measurement for 20 minutes using a constituent at pressure 2 and temperature of 170 degrees C of 100kg/cm, what was vulcanized at 200 degrees C for 4 hours was used as the test piece, and various measurement was presented. A result is shown in Tables 1-2.

[0029] Examples 1-3 are compounds (II) so that clearly from Tables 1-2. Even if it raises kneading ***** from 50 degrees C to 80 degrees C, a decrement of effective vulcanization torque is not seen, but there is also no aggravation of a compression set and a mechanical property, and it is stable, and the thermal stability of a compound is also good. On the other hand, since the substituent of p-grade of a phenolic compound is an electron-donative methoxy machine, the example 1 of a comparison is inferior in the thermal stability of a compound. Moreover, in the example 2 of a comparison, since it is a bisphenol compound, when raising kneading ***** from 50 degrees C to 80 degrees C, a fall of effective vulcanization torque and aggravation of a compression set happen, and it turns out that it is not desirable.

[0030]

[Table 1]

| | 実施例 1 | | 実施例 2 | | 実施例 3 | |
|--|-------------|----------|-------------|----------|----------------|----------|
| モノヒドロキシ化合物種類 | p-フェニルフェノール | | o-フェニルフェノール | | p-ヒドロキシ安息香酸メチル | |
| コンパウンド (I) 組成 (部) | | | | | | |
| フッ素ゴム | 100 | 100 | 100 | 100 | 100 | 100 |
| MT | 25 | 25 | 25 | 25 | 25 | 25 |
| MgO #150 | 3 | 3 | 3 | 3 | 3 | 3 |
| ビスフェノールA | - | - | - | - | - | - |
| p-メトキシフェノール | - | - | - | - | - | - |
| p-フェニルフェノール | 0.9 | 0.9 | - | - | - | - |
| o-フェニルフェノール | - | - | 0.9 | 0.9 | - | - |
| p-ヒドロキシ安息香酸メチル | - | - | - | - | 0.8 | 0.8 |
| コンパウンド (II) 組成 (部) | | | | | | |
| コンパウンド (I) | 128.9 | 128.9 | 128.9 | 128.9 | 128.8 | 128.8 |
| カルビット | 6 | 6 | 6 | 6 | 6 | 6 |
| TAIC | 3 | 3 | 3 | 3 | 3 | 3 |
| TBAHS | 1 | 1 | 1 | 1 | 1 | 1 |
| パーカドックス14 | 1 | 1 | 1 | 1 | 1 | 1 |
| 合計部数 | 139.9 | 139.9 | 139.9 | 139.9 | 139.8 | 139.8 |
| コンパウンド (II) 混練り条件 | | | | | | |
| 機種 | 4 インチロール | 4 インチロール | 4 インチロール | 4 インチロール | 4 インチロール | 4 インチロール |
| 表面設定温度 (℃) | 50 | 80 | 50 | 80 | 50 | 80 |
| 混練り時間 (分) | 5 | 5 | 5 | 5 | 5 | 5 |
| ムーニー粘度 (ML ₁₊₄ 、121℃) | 61 | 68 | 58 | 63 | 64 | 68 |
| (ML ₁₊₄ 、121℃) | 59 | 66 | 54 | 60 | 59 | 61 |
| 加硫挙動 (170℃) | | | | | | |
| t' (10) (分) | 1.65 | 1.42 | 1.54 | 1.57 | 1.47 | 1.68 |
| t' (90) (分) | 9.67 | 9.57 | 9.59 | 9.58 | 11.67 | 11.54 |
| M _L (dN-m) | 1.3 | 1.6 | 1.2 | 1.5 | 1.5 | 1.6 |
| M _B (dN-m) | 12.6 | 13.4 | 12.1 | 12.1 | 8.0 | 8.8 |
| M _B - M _L (dN-m) | 11.3 | 11.8 | 10.9 | 10.6 | 6.5 | 7.2 |
| 引張試験 | | | | | | |
| M ₁₀₀ (MPa) | 6.1 | 6.6 | 5.9 | 5.5 | 4.4 | 4.1 |
| T _B (MPa) | 16.1 | 15.5 | 16.4 | 15.4 | 11.4 | 11.9 |
| E _B (%) | 250 | 240 | 260 | 270 | 390 | 360 |
| 圧縮永久歪試験 (%) (175℃×70時間) | | | | | | |
| CS (%) | 29 | 23 | 30 | 24 | 47 | 39 |

[0031]

[Table 2]

| | 比較例 1 | | 比較例 2 | |
|--|---------------|---------------|---------------|---------------|
| モノヒドロキシ化合物種類 | ビスフェノール A | | p-メトキシフェノール | |
| コンパウンド (I) 組成 (部) | | | | |
| フッ素ゴム | 100 | 100 | 100 | 100 |
| MT | 25 | 25 | 25 | 25 |
| MgO #150 | 3 | 3 | 3 | 3 |
| ビスフェノールA | 0.6 | 0.6 | - | - |
| p-メトキシフェノール | - | - | 0.7 | 0.7 |
| p-フェニルフェノール | - | - | - | - |
| o-フェニルフェノール | - | - | - | - |
| p-ヒドロキシ安息香酸メチル | - | - | - | - |
| コンパウンド (II) 組成 (部) | | | | |
| コンパウンド (I) | 128.6 | 128.6 | 128.7 | 128.7 |
| カルビット | 6 | 6 | 6 | 6 |
| TAIC | 3 | 3 | 3 | 3 |
| TBAHS | 1 | 1 | 1 | 1 |
| パーカドックス14 | 1 | 1 | 1 | 1 |
| 合計部数 | 139.6 | 139.6 | 139.7 | 139.7 |
| コンパウンド (II) 混練り条件 | | | | |
| 機種 | 4 インチ チロール | 4 インチ チロール | 4 インチ チロール | 4 インチ チロール |
| 表面設定温度 (℃) | 50 | 80 | 50 | 80 |
| 混練り時間 (分) | 5 | 5 | 5 | 5 |
| ムーニー粘度 (ML ₁₊₁ 、121℃) | 66 | 89 | 61 | 67 |
| (ML ₁₊₁ 、121℃) | 80 | 130 | 61 | 67 |
| 加硫挙動 (170℃) | | | | |
| t' (10) (分) | 1.71 | 1.97 | 1.14 | 1.28 |
| t' (90) (分) | 8.96 | 11.85 | 9.42 | 9.94 |
| M _L (dN-m) | 1.5 | 1.6 | 1.5 | 2.3 |
| M _B (dN-m) | 17.4 | 10.6 | 14.1 | 15.2 |
| M _B - M _L (dN-m) | 15.9 | 9.0 | 12.6 | 12.9 |
| 引張試験 | | | | |
| M ₁₀₀ (MPa) | 8.5 | 5.5 | 7.6 | 8.8 |
| T _B (MPa) | 17.7 | 13.7 | 17.4 | 17.7 |
| E _B (%) | 200 | 230 | 220 | 200 |
| 圧縮永久歪試験 (%) (175℃×70時間) | | | | |
| CS (%) | 17 | 40 | 25 | 22 |

[0032]

[Effect of the Invention] The stability of the vulcanization behavior to kneading ***** is good, the fluororubber constituent of this invention has the good mechanical property, and it is useful on the radiator of an automobile, the O ring of engine ****, the gasket, the tube, the hose, etc.

[Translation done.]

JP 08-295726

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 CLAIMS

[Claim(s)]

[Claim 1] (A) fluoride -- a vinylidene -- and -- this -- copolymerization -- being possible -- one -- a sort -- more than -- others -- an ethylenic unsaturated monomer -- a copolymer -- **** -- becoming -- ** -- a fluorine -- elasticity -- a copolymer -- (-- B --) -- organic peroxide -- (-- C --) -- a unsaturated bond -- two or more pieces -- having -- bridge formation -- the hydroxide of an

assistant and (D) divalent metal or an oxide, (E) organic onium compound, and (F) polar compound -- since -- the fluororubber [Claim 2] (E) a component -- general formula R1 R2 R3 R4 N and SO3 R5 (R1 --) R2 And R3 The hydrocarbon group of 1 ** of the substitute of an identity or the different carbon numbers 1-20, or not replacing, R4 The hydrocarbon group of 1 ** of the substitute of a hydrogen atom or the carbon numbers 1-20, or not replacing, R5 Fluororubber constituent of the claim 1 which is the organic ammonium hydrogensulfate or the organic ammonium sulfonate shown by the hydrocarbon group of 1 ** of the substitute of a hydroxyl group or the carbon numbers 1-20, or not replacing.

[Claim 3] (E) The fluororubber constituent of the claim 1 which is hydroxylation organic ammonium in which a component is shown by general formula R6 R7 R8 R9 N and OH (R6, R7, R8, and R9 are the hydrocarbon group of 1 ** of the substitute of an identity or the different carbon numbers 1-20, or not replacing).

[Claim 4] (A) One fluororubber constituent of the claims 1-3 whose components are at least one sort chosen out of a fluoride vinylidene-tetrafluoroethylene-propylene system copolymer, a fluoride vinylidene-tetrafluoroethylene-hexafluoropropylene system copolymer, a fluoride vinylidene-hexafluoropropylene system copolymer, a fluoride vinylidene-tetrafluoroethylene-perfluoro-(methyl vinyl ether) system copolymer, and a fluoride vinylidene-tetrafluoroethylene-perfluoro-(propyl vinyl ether) system copolymer.

[Claim 5] (B) One fluororubber constituent of the claims 1-4 whose components are at least one sort chosen out of a peroxy ketal, dialkyl peroxide, diacyl peroxide, peroxy ester, and a hydroperoxide.

[Claim 6] (C) One fluororubber constituent of the claims 1-4 whose components are at least one sort chosen out of many allyl compounds, a dimethacrylate compound, a ***** compound, and a polybutadiene.

[Claim 7] (D) One fluororubber constituent of the claims 1-6 whose components are at least one sort of hydroxides and/or the oxide which are chosen out of magnesium, calcium, lead, and zinc.

[Claim 8] A component (E) Sulfuric-acid hydrogen tetrabutyl ammonium, sulfuric-acid hydrogen tetramethylammonium, A sulfuric-acid hydrogen tetraethylammonium, sulfuric-acid hydrogen trioctyl methylammonium, Sulfuric-acid hydrogen tridodecyl methylammonium, sulfuric-acid hydrogen trimethyl benzyl ammonium, Hydroxylation tetrabutyl ammonium, hydroxylation tetramethylammonium, A hydroxylation tetraethylammonium, hydroxylation trioctyl methylammonium, Hydroxylation tridodecyl methylammonium, trimethylbenzylammonium hydroxide, Tetrabutyl ammonium bromide, tetrabutyl ammonium chloride, Trimethyl benzyl ammonium bromide, 1, the p-toluenesulfonic-acid salt of 8-diazabicyclo [5.4.0] undeca-7-****, The fluororubber constituent of the claims 1, 4, 5, 6, or 7 which are at least one sort chosen out of the 8-benzyl -1, 8-diazabicyclo [5.4.0] undeca-7-***** chloride, tetrabutyl phosphonium chloride, and tetrabutyl phosphonium bromide.

[Claim 9] (F) One fluororubber constituent of the claims 1-8 whose components are at least one sort chosen out of water, a methanol, ethanol, propanol, i-propanol, n-butanol, s-butanol, t-butanol, a dimethyl sulfone, a diethyl sulfone, a sulfo run, an acetone, a methyl ethyl ketone, an acetylacetone, methyl acetate, ethyl acetate, and an ethyl malonate.

 [Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] this invention relates to the fluororubber constituent in which efficient organic peroxide vulcanization is possible.

[0002]

[Description of the Prior Art] The technique of carrying out copolymerization of the expensive monomer for organic peroxide vulcanization containing iodine or a bromine as the technique of vulcanizing a fluororubber by organic peroxide, the technique of performing an alkali treatment for a polymer to the bottom of presence of an onium salt, and enabling organic peroxide vulcanization, etc. are learned.

[0003] Moreover, the technique (JP, 1-198654, A) of cheap moreover enabling vulcanization by organic peroxide easily is learned by mixing the salt of unsaturation polyfunctional nature compounds, such as triallyl isocyanurate, the hydroxide of divalent metals, such as a calcium hydroxide, and an organic base, for example, diazabicycloundecen, for example. Furthermore, how (JP, 6-122801, A) to enable organic peroxide vulcanization by the few amount used compared with the case of an organic base is also learned by using an organic ammonium hydrogensulfate.

[0004]

[Problem(s) to be Solved by the Invention] Although the technique using the above-mentioned organic base was outstanding invention which does not need the special vulcanization site for organic peroxide vulcanization, in the organic base used there, it had comparatively much amount used required for organic peroxide vulcanization, therefore the problem was in the thermal resistance of a vulcanization moldings, and oil resistance. Moreover, from organic ammonium sulfuric-acid hydrogen, in order to acquire the physical properties by which the distributed status influenced vulcanizate nature and was stabilized, sufficient attention for the variance to a ** fluorine elastic copolymer needed to be paid.

[0005]

[Means for Solving the Problem] That the above-mentioned trouble should be solved, as a result of the study, this invention person improved the variance to the ** fluorine elastic copolymer of an organic onium compound by adding the polar compound which does not check vulcanization by organic peroxide, and found out that the stable vulcanizate physical properties without dispersion were acquired.

[0006] namely, the bridge formation which has the ** fluorine elastic copolymer which this invention becomes from the copolymer of (A) fluoride vinylidene (it is hereafter described as VdF) and this, and one or more sorts of other ethylenic unsaturated monomers that can be copolymerized, (B) organic peroxide, and two or more (C) unsaturated bonds -- the hydroxide of an assistant and (D) divalent metal or an oxide, (E) organic onium compound, and the fluororubber constituent characterized by consisting of a (F) polar compound are offered

[0007] (A) ** fluorine elastic copolymer consists of a copolymer of VdF and this, and one or more sorts of ethylenic unsaturated monomers that can be copolymerized in this invention. Here, as an ethylenic unsaturated monomer in which VdF and copolymerization are possible, perfluoro-, such as perfluoro-/perfluoro-/perfluoro-/olefins or fluoro olefins, such as hexafluoropropylene (it is hereafter described as HFP), a pentafluoro propylene, a propylene, triffe ***** ethylene a tetrafluoroethylene (it is hereafter described as TFE), a chlorotrifluoroethylene ethylene, and a fluoride vinyl, or / (methyl vinyl ether), / (propyl vinyl ether), and / (hexyl vinyl ether),] (alkyl vinyl ether) etc. is illustrated.

[0008] Among these, a VdF-TFE-propylene system copolymer, a VdF-TFE-HFP system copolymer, a VdF-HFP system copolymer, a VdF-TFE-perfluoro-(methyl vinyl ether) system copolymer, and a VdF-TFE-perfluoro-(propyl vinyl ether) system copolymer are adopted preferably.

[0009] Here, the copolymerization rate of VdF is suitably adopted by desired physical properties, the modality of ethylenic unsaturated monomer which carries out copolymerization, etc. Usually, what includes the polymerization unit of VdF at a rate beyond 0.1 mol % is adopted.

[0010] Since the vulcanization fluororubber excellent in engine-oil-proof nature is obtained when a VdF-TFE-propylene system copolymer is used especially, it is desirable. Especially, since the VdF-TFE-propylene system copolymer which includes the polymerization unit according to the polymerization unit according to the polymerization unit by VdF to 0.5-60 mol % and TFE to 20-60 mol % and a propylene at 20-50 mol % of a rate is excellent in the engine-oil-proof nature of a vulcanization fluororubber, a vulcanization property, and vulcanizate nature with a sufficient balance, it is desirable.

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oxide, an organic onium compound, a polar compound, and if needed

[0026] Such mixture is performed by a roll for rubber kneading, a kneader, or a Banbury mixer usually used from the former. Although especially the working condition at the time of mixture is not limited, the mixed variance of the above-mentioned compound can be enough carried out by usually kneading for about 10 - 60 minutes at the temperature of about 30-80 degrees C. Moreover, such a compound can be suitably scattered a melted part into a solvent, and it can also consider as a suspension solution. In addition, it is desirable to perform the work and operation at the time of mixture by selecting optimum conditions according to a use raw material, and the modality and the purpose of a compound.

[0027] The fabricating operation of the constituent of this invention may be carried out to the moldings like a sheet, a pipe, a rod, a tube, an angle, a channel, a rubber, and an application plate etc. by the continuous-molding processing methods, such as a knockout besides a usual golden die forming, transfer, a roll coat, brushing, and sinking in. In addition, a fabricating operation may be carried out to section-shape and special mold-goods, for example, sponge, -like rubber etc. by various fabricating-operation methods. Thus, the after-mentioned unities the constituent of this invention by which the fabricating operation was carried out, it is made vulcanizate by the vulcanization means and a vulcanization rubber goods is obtained by it.

[0028] In this invention, vulcanization operation can adopt the operation usually used from the former. For example, the operation heated while pressurizing in a form block is adopted, and after extruding and fabricating by the calendering roll etc., the operation heated in a heating furnace or a steamy iron pot may be adopted.

[0029] As for the working condition at the time of vulcanization etc., it is desirable to carry out by selecting optimum conditions according to a use raw material or combination. As for the temperature at the time of vulcanization, about 60-250-degree C about 120-200 degrees C are usually adopted preferably. Moreover, although especially a heating time is not limited, according to the modality of organic peroxide, the domain of it is 1 minute - 3 hours, and it is preferably selected within the limits of 5 minutes - 2 hours. A heating time can be shortened if heating temperature is made high.

[0030] In addition, re-heating processing of the vulcanizate obtained can also be adopted and an effect is in the enhancement in a physical property. For example, it is 180-230 degrees C in temperature preferably, and 150-250 degrees C of re-heating processings of about 2 - 25 hours may be adopted.

[0031]

[Function] As for the constituent which blended the polar compound in order to raise the dispersibility of an organic onium compound, the outstanding vulcanizate nature which dispersion does not have is obtained by organic peroxide vulcanization. Moreover, the organic onium compound in this invention is effectively conjectured to become good [the thermal resistance or oil resistance of vulcanizate which organic peroxide vulcanization advances and are obtained] by the comparatively little amount used compared with the organic base used conventionally.

[0032]

[Example] Examples 1-8 are examples and Examples 9-11 are examples of a comparison.

[0033] According to the composition (weight section) shown in Table 1 and Table 2, the rubber constituent which mixed various kinds of charges of a compounding agent for 10 minutes with two rolls, and was mixed sufficiently uniformly to the grade from which an organic onium compound is not checked visually was obtained. After carrying out press cure of them for 10 minutes at 170 degrees C, it vulcanized secondarily at 230 degrees C in oven for 24 hours.

[0034] According to JIS-K6301, the ordinary state physical properties of the obtained vulcanizate, thermal resistance (230 degree-Cx 70 hours), and oil resistance (oil : ***** CASL clean SG, 175 degree-Cx 70 hours) were measured. A result is shown in Table 3 and Table 4 (M100 is 100% modulus).

[0035] In addition, each fluororubber of Table 1 and Table 2 is what was obtained by the emulsion-polymerization method, and shows the polymerization unit inclusion rate (mole ratio) by each of those monomers in Table 5. Moreover, other cable addresses in Tables 1 and 2 etc. are shown in Table 6.

[0036]

[Table 1]

| | 例1 | 例2 | 例3 | 例4 | 例5 | 例6 | 例7 | 例8 |
|-----------|-----|-----|-----|-----|-----|-----|-----|-----|
| フッ素ゴム-1 | 100 | 100 | 100 | | | | | |
| フッ素ゴム-2 | | | | 100 | 100 | | | |
| フッ素ゴム-3 | | | | | | 100 | 100 | 100 |
| MTカーボン | 25 | 25 | 25 | 25 | 25 | 25 | 25 | 25 |
| キョーワマグ150 | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| TAIC | 3 | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| パーカドックス14 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| TBAHS | 0.8 | | 0.8 | | | 0.5 | | |
| TBAOH | | 1 | | | 1 | | | |
| DBU | | | | 0.7 | | | | |
| TBAB | | | | | | | 3 | |
| TPBPC | | | | | | | | 3 |
| カルビット | 3 | 3 | 3 | 1.5 | | 1.5 | | |
| 水 | 1.5 | | | | | | | |
| メタノール | | 1.5 | | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |
| スルホラン | | | 1 | | | | | |

[0037]

[Table 2]

| | 例9 | 例10 | 例11 |
|-----------|-----|-----|-----|
| フッ素ゴム-1 | 100 | | |
| フッ素ゴム-2 | | 100 | |
| フッ素ゴム-3 | | | 100 |
| MTカーボン | 25 | 25 | 25 |
| キョーワマグ150 | 3 | 3 | 3 |
| TAIC | 3 | 3 | 3 |
| パーカドックス14 | 1 | 1 | 1 |
| TBAHS | 0.8 | 0.5 | 0.5 |
| カルビット | 3 | 1.5 | 1.5 |

[0038]

[Table 3]

| | 例 1 | 例 2 | 例 3 | 例 4 | 例 5 | 例 6 | 例 7 | 例 8 |
|---|-----|-----|-----|-----|-----|-----|-----|-----|
| 硬さ (JIS-A) | 75 | 74 | 75 | 70 | 75 | 76 | 73 | 74 |
| M ₁₀₀ (kgf/cm ²) | 72 | 72 | 71 | 60 | 74 | 82 | 62 | 59 |
| 引張強さ (kgf/cm ²) | 218 | 207 | 200 | 200 | 205 | 190 | 170 | 165 |
| 伸び (%) | 250 | 290 | 250 | 270 | 220 | 180 | 170 | 195 |
| 圧縮永久歪 (%) | 21 | 27 | 21 | 25 | 20 | 31 | 38 | 37 |
| 耐熱性 | | | | | | | | |
| 硬さ変化 (度) | 2 | 1 | 2 | 1 | 1 | 1 | 2 | 3 |
| 引張強さ変化率 (%) | -2 | -6 | -3 | -12 | -9 | -2 | -12 | -8 |
| 伸び変化率 (%) | 4 | -6 | -1 | -7 | -5 | -2 | -5 | -4 |
| 耐油性 | | | | | | | | |
| 硬さ変化 (度) | -7 | -5 | -7 | -3 | -4 | -1 | -6 | -8 |
| 引張強さ変化率 (%) | -10 | -11 | -10 | -18 | -18 | -21 | -29 | -30 |
| 伸び変化率 (%) | 0 | -5 | 5 | 4 | -10 | -25 | -30 | -31 |
| 体積変化率 (%) | 7 | 8 | 8 | 5 | 4 | 2 | 6 | 8 |

[0039]

[Table 4]

| | 例 9 | 例10 | 例11 |
|---|-----|-----|-----|
| 硬さ (JIS-A) | 70 | 70 | 73 |
| M ₁₀₀ (kgf/cm ²) | 35 | 54 | 59 |
| 引張強さ (kgf/cm ²) | 199 | 190 | 180 |
| 伸び (%) | 310 | 270 | 190 |
| 圧縮永久歪 (%) | 26 | 30 | 35 |
| 耐熱性 | | | |
| 硬さ変化 (度) | 2 | 3 | 2 |
| 引張強さ変化率 (%) | -8 | -1 | -5 |
| 伸び変化率 (%) | 7 | 6 | -7 |
| 耐油性 | | | |
| 硬さ変化 (度) | -2 | -1 | -2 |
| 引張強さ変化率 (%) | -16 | -22 | -21 |
| 伸び変化率 (%) | 4 | 11 | 14 |
| 体積変化率 (%) | 9 | 7 | -30 |

[0040]

[Table 5]

| | |
|---------|------------------------|
| フッ素ゴム-1 | VdF/TFE/プロピレン= 4/52/44 |
| フッ素ゴム-2 | VdF/TFE/プロピレン=35/40/25 |
| フッ素ゴム-3 | VdF/TFE/HFP =60/15/25 |

[0041]

[Table 6]

| | |
|-----------|--|
| キョーワマグ150 | 酸化マグネシウム (協和化学工業製) |
| TAIC | トリアリルシアヌレート |
| パーカドックス14 | α 、 α' -ビス (t-ブチルペルオキシ) -p-ジ イソプロピルベンゼン (日本油脂製) |
| TBAHS | 硫酸水素テトラブチルアンモニウム |
| TBAOH | 水酸化テトラブチルアンモニウム |
| DBU | 1,8-ジアザビシクロ [5.4.0] ウンデカ- 7-エンのp-トルエンスルホン酸塩 |
| TBAB | テトラブチルアンモニウムブロミド |
| TPBPC | トリフェニルベンジルホスホニウムクロリド |
| カルビット | 水酸化カルシウム (近江化学製) |

[0042]

[Effect of the Invention] Efficient organic peroxide vulcanization is possible for the fluororubber constituent of this invention, and, moreover, the stable vulcanizate nature without dispersion is obtained. Furthermore, the radiator of an automobile, the O ring of the circumference of an engine, a gasket, the sealant, the diaphragm, the tube, the hose, etc. are [in / wide range intended use such as the same parts, such as a food plant and a chemical processing plant, / based on the vulcanizate nature which was excellent in various] very useful.

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CLAIMS

[Claim(s)]

[Claim 1] The polymer frame-common-equipment bridge assistant characterized by consisting of a diaryl isocyanurate salt.

[Claim 2] the bridge formation according to claim 1 whose polymer is one sort chosen from the group of ethylene-propylene-diene rubber, chlorinated-polyethylene rubber, chlorosulfonated polyethylene rubber, hydrogenation acrylonitrile-butadiene rubber, a fluororubber, ethylene-vinyl acetate rubber, polyethylene, polypropylene, a fluororesin, vinyl chloride resin, and polystyrene -- an assistant

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 DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the polymer frame-common-equipment bridge assistant excellent in the thermal stability at the time of a manipulation in detail about a polymer frame-common-equipment bridge assistant.

[0002]

[Description of the Prior Art] the bridge formation which has an allyl group -- by the combined use with a cross linking agent, or the combined use with actinic-rays irradiation processing of an electron ray etc., an assistant raises the crosslinking density of a polymer and improves the thermal resistance of rubber, a resin, etc., weatherability, an electrical property, etc. For example, triallyl isocyanurate improves the thermal resistance of various polymers with the thermal resistance which was excellent in the triazine ring used as the skeleton, and the mechanical property of various polymers, weatherability, a adding-water-proof resolvability, etc. improve it with the unique structure and unique property.

[0003]

[Problem(s) to be Solved by the Invention] by the way -- comparatively -- an elevated temperature -- bridge formation -- cases, such as the polymer which processes, for example, a fluororesin etc., -- bridge formation -- the bridge formation added by scattering loss of an assistant -- an assistant does not function in many cases effectively this invention is made in view of ** or *****, and the purpose is in offering the polymer frame-common-equipment bridge assistant which was excellent in the thermal stability so that it might not vaporize at the time of a hot working.

[0004]

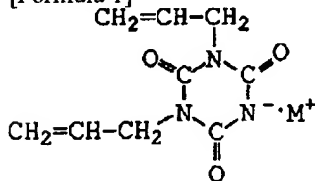
[Means for Solving the Problem] That is, the summary of this invention consists in the polymer frame-common-equipment bridge assistant characterized by consisting of a diaryl isocyanurate salt.

[0005]

[Embodiments of the Invention] Hereafter, this invention is explained in detail. this invention -- setting -- bridge formation -- the diaryl isocyanurate salt suitably used as an assistant is expressed with the following general formula

[0006]

[Formula 1]



[0007] M expresses a monovalent metal, for example, Li, Na, K, etc., among the above-mentioned general formula. A suitable monovalent metal is Na.

[0008] In this invention, a diaryl isocyanurate salt may be a salt which two or more diaryl isocyanurate units combined to the metal more than a bivalency. For example, as a bivalent metal, Be, Mg, calcium, Sr, Ba, Zn, Cd, etc. are mentioned. The diaryl isocyanurate salt of what ** is also powder. The most desirable diaryl isocyanurate salt is calcium salt or Zn salt of diaryl isocyanurate.

[0009] the monovalent metal salt of diaryl isocyanurate -- for example, a neutralization with diaryl isocyanurate and the aqueous solution of a metal hydroxide -- moreover, the bivalent metal salt of diaryl isocyanurate -- for example, it can obtain easily by the double decomposition reaction of a monovalent metal salt and a bivalent metal chloride

[0010] bridge formation of this invention -- although especially the polymer to which an assistant is applied is not limited, it is one sort chosen from the group of ethylene-propylene-diene rubber, chlorinated-polyethylene rubber, chlorosulfonated polyethylene rubber, hydrogenation acrylonitrile-butadiene rubber, a fluororubber, ethylene-vinyl acetate rubber, polyethylene, polypropylene, a fluororesin, vinyl chloride resin, and polystyrene as a suitable polymer Vulcanization and bridge formation are possible for these polymers by actinic-rays irradiation nature processing of addition or an electron ray of organic peroxide etc.

[0011] the bridge formation which consists of the diaryl isocyanurate salt of this invention -- an assistant -- usually -- two pieces or four allyl groups -- being based -- bridge formation of two organic functions or four organic functions -- as an assistant, it acts

and the physical properties of a polymer are improved and bridge formation of this invention -- according to well-known technique, by the combined use with the cross linking agent which was [amine / organic peroxide,] suitable, or the combined use with actinic-rays processing of an electron ray etc., an assistant raises the crosslinking density of a polymer and improves the thermal resistance of rubber, a resin, etc., weatherability, an electrical property, etc.

[0012] bridge formation of this invention -- although the amount of the assistant used can be chosen from a large area, it is alike and receives a polymer and is usually preferably chosen from 1 - 10% of the weight of a domain 0.1 to 20% of the weight by the physical-properties enhancement made into the modality and the purpose of a polymer bridge formation of this invention -- mixed processing with an assistant and a polymer is performed using a twin screw extruder, a Banbury mixer, a Brabender, etc., and, as for processing temperature, temperature high [beyond the melting point of a polymer] is adopted

[0013] In the above-mentioned mixed processing, a well-known additive can be used suitably. Typically as such an additive, the antioxidant which consists of alkylation phenols, such as "***** NOx 1010", the "***** NOx 1015", and the "***** NOx 1076", is mentioned. A flame retarder, a pigment, etc. are mentioned to others. bridge formation of this invention -- according to the assistant, a working temperature is high conventionally -- elapsing -- bridge formation -- effective crosslinking reaction can be performed to the polymer which was not effectively used in an assistant

[0014] As organic peroxide, 2, the hydroperoxides like 5-dihydroxy peroxide, and the dialkyl allyl-compound peroxide like dicumyl peroxide are mentioned, for example. In the case of a fluororesin like the copolymer of ethylene and a tetrafluoroethylene, a bridge is constructed by actinic-rays irradiation nature processing. in order that quantity of radiation may avoid a degradation of ***** by superfluous irradiation -- general -- about 50 -- you may be M rads or less although it depends for concrete dosage on the target degree of cross linking -- usually -- 2-40 -- M rads are preferably chosen from the domain of 3-20M rad Although irradiation of an actinic rays is generally performed near a room temperature, you may irradiate at the elevated temperature beyond it.

[0015]

[Example] Hereafter, although an example explains this invention still in detail, this invention is not limited to the following examples, unless the summary is exceeded.

[0016] ETFE (ethylene --) of the examples 1-3 and 270 degrees C of the example 1 (1) melting points of a comparison The fluororesin which consists of the hard segment which has a tetrafluoroethylene and a hexafluoropropylene unit, (2) -- TPE ("-- the Daikin Industries "***** T-530":vinylidene full ora id --) The thermoplastic elastomer which consists of the soft segment which has a tetrafluoroethylene and a hexafluoropropylene unit, (3) A diaryl isocyanurate salt and (4) antioxidants (product made from Ciba-Geigy "***** NOx 100") were used, and mixed processing was carried out at 300 degrees C by the 1.5 inch 1 shaft screw extruder as preparation composition given in Table 1. each obtained resin constituent -- the slab of 25 mills -- fabricating -- an electron ray -- amount of whole line 5M rad -- irradiating -- bridge formation -- after processing, physical-properties measurement was presented A result is shown in Table 1 and 2.

[0017] Measurement of tensile strength and a ***** modulus (flexibility) was performed in each sample which performed after [electron ray un-irradiating, an electron beam irradiation, and an electron beam irradiation] annealing processing. E30 (force / initial cross section required in order to attain elongation 30%), and M100 (force / initial cross section required in order to attain elongation 100%) were performed in each sample which performed after [an electron beam irradiation and an electron beam irradiation] annealing processing. Solvent resistance, heat aging nature, and the low-temperature impact-brittleness heat test were performed only in the electron-beam-irradiation sample.

[0018] Measurement of E30 and M100 was performed according to the static modulus method of ** erection *****
***** (The Electrical Manufacturer) by R **** black (R, M, Black).

[0019] Measurement of the tensile strength and elongation which include the measurement performed after heat aging and solvent immersing is ASTM. D It carries out according to 414 and measurement of a ***** modulus is ASTM. D It carries out according to 882 and measurement of a low-temperature impact-brittleness heat test is ASTM. It carried out according to D746.

[0020] The resistance to solvents test was flooded with each liquid in the test sample (thickness of 0.025 inches, width of face of 1/8 inch), namely, it was immersed at the room temperature at "the ***** roll (Skydrol) 500" (Monsanto Co. make), it was immersed in "DS-2" at the room temperature for 4 hours for 24 hours, and, subsequently it carried out by measuring tensile strength and elongation. "The ***** roll (Skydrol) 500" is a phosphate ester hydraulic medium, and "DS-2" is the mixture of a diethylenetriamine (70%), an ethylene glycol monoester (28%), and NaOH (2%).

[0021] Into the kiln which adjusted the test sample (thickness of 0.025 inches, width of face of 1/8 inch) at 250 degrees C, the heat aging examination was performed tensile strength (T) and by being extended and measuring (E), respectively, one week and after leaving it for two weeks.

[0022]

[Table 1]

***** An example The example of a comparison 1 2 3 1 <Combination composition> ETFE 77 77 77 77TPEs 20 20 20 20 antioxidants 0.9 0.9 0.9 A 0.9 ** ** allyl-compound isocyanurate calcium salt 3 -- - ** ** Allyl-Compound Isocyanurate Zn Salt - 3 - - ** ** Allyl-Compound Isocyanurate Aluminum Salt - - 3- ** ** allyl-compound isocyanurate - - - 3 <tensile strength (Kg/cm2)> electron ray un-irradiating. 340 350 345 272 electron beam irradiations 336 390 360 260 electron beam irradiations / annealing 343 382 358 265 <elongation (%)> electron ray un-irradiating. 315 320 320 400 electron beam irradiations 240250 245 260 electron beam irradiations / annealing 220 225 225 220 <2% scan ***** (Kg/cm2)> electron ray un-irradiating. 4126 4200 4188

4100 electron beam irradiations 5381 5521 5400 5110 electron beam irradiation / annealing 4200 4300 4250
4050***** [0023]

[Table 2]

***** An example The example of a
comparison 1 2 3 1 <E30 (300 **) (Kg/cm2) > electron beam irradiation 8.3 9 8.8 6 electron beam irradiations / annealing 11.0
12 11.5 10<E100 (300 degrees C) (Kg/cm2) > electron beam irradiation 5.05.5 5.5 3 electron beam irradiations / annealing 6.9
6.5 6.6 5 <solvent resistance (Kg/cm2)> T/EDS-2 (24 time / room temperature) 395/21 400/25 401/24 300 / 35 ***** **
roll 500 (24 time / room temperature) 348/16 360/17 355/20 277/30 <heat aging nature (Kg/cm2)> One week (250 degrees C)
of T/E 294/14.5 311/15 300/15 255/302 week (250 degrees C) 276/12 301/14 288/14 250/28 <low-temperature
impact-brittleness temperature> (it is success at -65 degrees C altogether)

***** [0024]

[Effect of the Invention] according to this invention explained above, the polymer frame-common-equipment bridge assistant
which was excellent in the thermal stability so that it might not vaporize at the time of a hot working is offered -- having -- the
polymer frame-common-equipment bridge assistant of this invention -- especially -- high temperature -- bridge formation -- it is
suitable as an object for fluororesins processed

[Translation done.]